PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

in re Application of:

John R. Walton, Scott W. Duggan, and

Michael R. Fagan

Application No.: 09/932,042

Filing Date: August 17, 2001

Confirmation No.: 4497

Group Art Unit: 1754

Examiner: Chester T. Barry

A Method for Reducing Hydrogen Sulfide Emissions from Wastewater

EXPRESS MAIL LABEL NO: US DATE OF DEPOSIT: September xx, 2003

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Sir:

DECLARATION OF JOHN R. WALTON PURSUANT TO 37 CFR § 1.132

- I, John R. Walton, being duly warned that willful false statements and the like are punishable by fine or imprisonment or both, under 18 U.S.C. § 1001, and may jeopardize the validity of the patent application or any patent issuing thereon, state and declare as follows:
- 1. All statements herein made of my knowledge are true and statements made on information or belief are believed to be true. The Exhibits attached hereto are incorporated herein by reference.
- 2. I have more than 20 years experience in the use of chemical oxidation technologies for environmental processes, including ten years as the chief technologist for the leading

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multi-product chemical service firms that specialize in the field. Through this period, I have

managed or overseen more than 75 projects related to the design and operation of odor

(sulfide) control processes for both municipalities (collection and treatment of water and

wastewater) and industry (sour waters/sludges from petroleum operations, steel production,

paper processing, food processing, geothermal operations, and chemical manufacture). This

technical background establishes my expertise in the field of wastewater treatment. My

curriculum vitae is attached hereto as Exhibit A.

3. I have read and am familiar with the contents of the above-referenced patent

application. I further understand that the nature of the rejection at issue in the pending

application is that the Examiner believes that the present invention is taught by Pomeroy et

al., Sewage Works, July 1946, pp. 597-640 ("Pomeroy"). The purpose of this declaration is

to address this issue.

Pomeroy describes the addition of ferrous sulfate to a wastewater stream within a

sanitary sewer. On page 628 of the Pomeroy reference, the natural aeration of sewage to

which a ferrous salt has been added is described.

In contrast to Pomeroy, the addition of the oxident to the wastewater stream according

to the present invention is deliberate. The natural aeration of the sewage described by

Pomeroy is insufficient to regenerate iron ions in an amount sufficient to demonstrate a

significant beneficial effect downstream of the point of oxidant addition in a wastewater

treatment system.

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The methods of the invention have been applied to a large municipal sewage interceptor, the Miller-Holder Interceptor, Orange County Sanitation Districts (Fountain Valley, CA). The Miller-Holder interceptor is a long, high sulfide-producing sewer that discharges into Plant No. 2 of the Orange County Sanitation Districts. The interceptor is a free-flowing gravity reinforced concrete pipe with average velocity 2.5 feet per second (fps) and diameters increasing from 48" (at the upper reaches) to 78" (at the treatment plant). It gathers about 24 million gallons per day (MGD) over 23 miles with a transit time from the uppermost reaches to the treatment plant of about 13 hours.

The results of the field test demonstrated that the utilization efficiency of iron for sulfide removal is greatly improved by adding H_2O_2 (as opposed to allowing for natural aeration only). Without any addition of chemicals, the dissolved sulfide levels increase along the interceptor such that, from the midpoint to the treatment plant, the average level is about 10 - 12 mg/L. As additional flows are added, the sulfide loading increases from about 1000 lbs-Sulfide per day (at the treatment plant):

Sulfide loading = $2,000 \text{ lbs / day} = (10 \text{ mg/L sulfide}) \times (1-\text{lb / 454,000 mg}) \times (3,785,000 \text{ liters / 1-MG}) \times (24 \text{ MGD}).$

Ferrous chloride solution (36% w/w FeCl₂) was then added at a rate of 960 gallons per day (gpd) at the top of the interceptor. This equates to a feed rate of 1,786 lbs-Fe per day (specific gravity = 1.41, giving 1.86 lbs-Fe per gallon 36% w/w FeCl₂), and corresponds to a dose ratio of 0.90 lbs-Fe per lb-Sulfide, or 55% of theory (theory = 1.64 lbs-Fe²⁺ per lb-Sulfide). The results show dissolved sulfide levels at the midpoint and treatment plant averaged about 0.5 mg/L and 4.2 mg/L, respectively. This equates to a removal of 5.8 mg/L (or 1,160 lbs-Sulfide per day) at the treatment plant, for a reduction of 58%:

Sulfide removed = 1,160 lbs / day =

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(10.0 – 4.2 mg/L) x (1-lb / 454,000 mg) x (3,785,000 liters / 1-MG) x (24 MGD). This represents an effective removal rate of 0.65 lbs-Sulfide per lb-Fe, or inversely, an iron utilization efficiency of 1.54 lbs-Fe per lb-Sulfide removed. This is 6% above theory, indicating only marginal benefit afforded by natural aeration of the sewage, as is predicted by conventional engineering models for such large diameter, low velocity sewers. See Exhibit B (providing a graph of natural aeration rates in sewers flowing half full). Given that typical velocities are 2-3 fps, the graph shows that the re-aeration factor for large diameter interceptors is < 0.05 while that for smaller diameter tributaries is > 1 - 1.5. This is a difference of 20 – 30 times. Hence, the value of adding a chemical oxidant to achieve a degree of iron regeneration simply is not practical by way of natural re-aeration alone.

In the third phase of the field test, H_2O_2 (50% w/w) was added at a rate of 325 gpd at the midpoint along the interceptor, in addition to the 960 gpd of 36% w/w FeCl₂ added at the top of the interceptor. This represents a feed rate of 1625 lbs- H_2O_2 per day (specific gravity = 1.20, 5.0 lbs- H_2O_2 per gallon). The results show dissolved sulfide levels at the treatment plant averaged about 0.5 mg/L, which equates to a removal of 9.5 mg/L (or 1,900 lbs-Sulfide per day), for a reduction of 95%:

Sulfide removed = 1,900 lbs / day = $(10.0 - 0.5 \text{ mg/L}) \times (1-\text{lb} / 454,000 \text{ mg}) \times (3,785,000 \text{ liters} / 1-MG) \times (24 \text{ MGD}).$

This represents an effective removal rate of 1.06 lbs-Sulfide per lb-Fe, or inversely, an iron utilization efficiency of 0.94 lbs-Fe per lb-Sulfide removed. This is 74% above theory, indicating a substantial benefit afforded by the deliberate addition of H_2O_2 . Thus, the addition of approximately 1625 pounds hydrogen peroxide at the interceptor midpoint in conjunction with the ferrous ion addition reduced the sulfide mass loading at the interceptor terminus to approximately 100 pounds per day.

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In short, application of the present invention provided an effective control ratio of ferrous ion to sulfide 45% less than that predicted by theory. Accordingly, the practical benefits of the present invention include a substantial reduction in the detrimental effects resulting from the addition of ferrous ion, a 45% reduction in solids production at the treatment plant, 45% less salinity, and 45% less alkalinity.

5. In addition to an improvement in the utilization efficiency of iron for sulfide removal by addition of H₂O₂ as opposed to allowing for natural aeration only, the conversion of ferrous iron to ferric iron by the deliberate addition of H₂O₂ is rapid, occurring within minutes as opposed to allowing for natural aeration only.

This series of tests involved quantifying the ferrous and ferric iron levels entering the treatment plant with and without the deliberate addition of H₂O₂ (384 gpd of 50% w/w H₂O₂, or 1,920 lbs-H₂O₂ per day). Table 1 shows that, without the addition of H₂O₂, the ratio of Fe²⁺ to Fe³⁺ is about 80:20, indicating only marginal conversion provided by natural aeration within the interceptor and treatment plant headworks.

Table 1. Ferrous and ferric iron levels entering the treatment plant without H₂O₂ added (expressed as mg/L Fe).

	4/21/2000			4/22/2009			4/28/2008		
Time	Pa ^{ra} ju	Pe ^{pe}	Po ^{Tube}	Po	Fo	Fe ^{Telal}	Feb	Par	Fe ^{Trick}
9:00	79	1.3	2	7.2	24	44	7.8	0.1	7.0
11:00	8.4	2.0	8.6	4.2	2.1	4	6.2	ม	8.4
18:00	4.7	2.6	7.3	6.3	1.1	ũ	ü	2.7	4
15:00	72	1.2	6.3	8.6	8.8	2.5	6.7	1.7	ㅂ
	_			_	_		_	<u> </u>	_
Averege	6.6	1.8	8.3	44	1.8	ا مه	6.6	1.7	8.6
	79%	21%	ļ	40%	20%		80%	200	

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Table 2 shows that, with the addition of H_2O_2 , the ratio of Fe^{2+} to Fe^{3+} shifts to about 20:80, which indicates a substantial conversion of ferrous to ferric iron. These results reflect a reaction time of about 5 minutes from the point of H_2O_2 addition to the point of sampling.

Table 2. Ferrous and ferric iron levels entering the treatment plant with H₂O₂ added. (expressed as mg/L Fe).

	6/20/2008				6/21/2003	
Time	Fe ³⁴	Fe ³⁺	Fe ^{Teld}	Fe ²⁺	Fe ³⁺	Fe ^{7ccd}
7:00	2.3	6.2	8.6	0.8	7.5	8.4
8:30	3.0	7.0	10.0	0.8	6.8	7.8
10:00	0.0	6.2	7.2	0.9	8.4	8.3
11:05	1.3	7.0	8.3	2.8	7.3	10.0
12:30	2.5	6.7	9.2	2.9	10.2	13.1
13:46	1.6	6.2	7.8		10.2	19-1
f						
Average	2.0	6.6	8.5	1.6	8.0	9.7
	23%	77%		17%	83%	•••

Samples also were collected 10-15 minutes prior to the treatment plant (and the point of H_2O_2 addition) and analyzed for Fe^{2+} to Fe^{3+} levels. These data (see Table 3) show an even higher ratio of Fe^{2+} to Fe^{3+} (85-90 : 10-15) before H_2O_2 addition, indicating even lesser conversion of Fe^{2+} to Fe^{3+} by natural aeration within the interceptor.

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Table 3. Ferrous and ferric iron levels before and after H₂O₂ addition (expressed as mg/L Fe).

	Time	Sampling Location 10-15 minutes Before H2O2 Injection			Sampling Location 5 minutes After H2O2 Injection		
Date		Per	Pe ^{Se}	Fig. Total	Ps ²⁰	Fe ³⁴	Fe ^{Total}
5/20/2009	7:00	7.6	1.4	8.9	2.3	6.2	8.5
	6:30	9.3	0.4	9.8	1.0	7.0	10.0
	10:00	7.8	1.0	8.8	0.0	8.2	7.2
	11:06	8.3	0.8	8.1	1.3	7.0	8.3
	12:30	8.6	1.4	10.9	2.6	6.7	9.2
	13:45	8.6	1.3	2.0	1.6	8.2	7.8
		_	_				
	average	6.6	1.0	9.5	2.0	6.6	8.5
		80%	11%		22%	77%	
6/21/2003	9:00	6.2	0.2	6.6	0.8	7.5	8.4
	10:00	8.1	2.7	11.7	0.8	6.3	7.5
	12:45		•		0.9	8.4	8.3
	13:46	8.3	1.4	9.7	2.8	7.3	10.0
	16:00	10.9	1.8	12.7	2.9	10.2	18.1
				<u> </u>			-
	Average	8.6	1.6	10.1	1.6	8.0	9.7
		05%	18%		17%	89%	

As these experimental results show, the conversion of ferrous iron to ferric iron by the deliberate addition of H_2O_2 is rapid, occurring within minutes, as opposed to allowing for natural aeration only.

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6. In short, the present invention is distinguishable over the methods taught by Pomeroy.

Nowhere in Pomeroy is the deliberate addition of an oxidant to wastewater to which iron has been added upstream described. As demonstrated herein, the methods of the present invention provide substantial benefit to treatment of wastewater over previously known treatment methods.

Date:

9-8-2003

John R. Walton

Attachments

Exhibits A and B

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EXHIBIT A
Curriculum Vitae

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John R. Walton Peroxide, Inc.

1 7 2003

Vice President, Technology and Program Development

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I have more than 20 years experience in the use of chemical oxidation technologies for environmental processes, including ten years as the chief technologist for the leading multi-product chemical service firms that specialize in the field. Through this period, I have managed or overseen more than 75 projects related to the design and operation of odor (sulfide) control processes for both municipalities (collection and treatment of water and wastewater) and industry (sour waters/sludges from petroleum operations, steel production, paper processing, food processing, geothermal operations, and chemical manufacture).

Education

Graduate - Business Administration, University of Houston (9/82 - 5/84). Credits completed: 21 of 36 (Night-school. MBA Program). Degree program was abandoned due to the increasing demands of international business travel.

M.S. - Environmental Sciences, University of Texas (10/78 - 3/81) Specialization: Water Quality.
 Thesis: Environmental fate of selected industrial chemicals.

B.S. - Biochemistry / Microbiology, University of Houston (8/75 - 8/78). Specialization: Environmental Microbiology.

Work Experience

8/96 - present <u>US Peroxide, Inc.</u> Serve as V.P., Technology and Program Development for this specialty chemical services firm, and manage Western operations for applying hydrogen

peroxide (and related chemicals) for treating drinking water, wastewater, and hazardous

wastes, with an emphasis on developing new applications and technologies.

See: http://www.h2o2.com/

1/93 - 8/96 <u>Vulcan Chemical Technologies (formerly Peroxidation Systems, Inc).</u> Directed the

firm's chemical services business, including its Sulfide Control Service (for municipal water and wastewater treatment) and Chlorine Dioxide Service (for drinking water treatment). Led diversification of the firm's product and service capabilities, including

nationwide

expansion.

1/90 - 1/93 Carus Chemical. Led the firm's chemical oxidation services for wastewater treatment

(ChemOx Environmental Services), employing technologies such as ozone, hydrogen peroxide, potassium permanganate, chlorine dioxide, UV photolysis, and O₁-H₂O₂ in both

the municipal and industrial sectors.

9/80 - 1/90 Solvay-Interox (manufacturer of hydrogen peroxide). Joined the firm as an Applications

Chemist/Engineer, and resigned 10 years later as Business Manager, Environmental and

Chemical Processing. Further detail follows.

9/80 - 9/84

Applications Chemist/Engineer. Areas of work included wastewater odor control, conditioning and stabilization of waste sludges, treatment of phenol-, sulfide-, and cyanide laden wastes, enhancement of biological treatment systems, development of Advanced Oxidation Technologies (ozone/hydrogen peroxide and UV catalyzed H_2O_2), and stabilization of H_2O_2 solutions.

9/84 - 6/86

<u>Technical Services Engineer</u>. Assisted new customers in the design and installation of H_2O_2 storage/dosing systems for both the chemical processing and environmental industries. Developed customized stabilizer formulations that improved the cost-performance of the company's products.

6/86 - 12/89

Development Manager. Led a team charged with identifying and pursuing commercial ventures pertaining to peroxygen chemistry. Key functions included business negotiations and subsequent liaison with the R&D, engineering and business groups of other chemical producers and independent research firms to develop new chemical oxidation technologies, including the processes and products derived therefrom.

12/89 - 1/90

Business Manager. Managed hydrogen peroxide sales efforts to the environmental and chemical processing industries, including related business development activities (toll processing, custom synthesis, process development).

5/79 - 9/80

University of Texas Health Science Center at Houston. Graduate Research Asst. charged with maintaining and coordinating a water quality laboratory, which involved consulting on biosystems operation for a number of regional chemical firms.

Professional

Society Memberships:

American Chemical Society, Water Environment Federation, American Water

Works Association

Management Training:

One of twenty employees (selected from a pool ca. 1000) chosen to participate in Solvay's accelerated management training program, which involved ca. 200

hours per year for a three year period.

Abstracts of Work

Thesis:

"Toxicology and Fate of Selected industrial Chemicals in Aquatic Ecosystems", Univ. of Texas, School of Public Health, March 1981. Dr. Ernst M. Davis,

principal advisor.

Patents:

USP 4,569,769 USP 5,200,092 USP 5,336,431

Publications and Papers:

- 1. "Impact of Coal Ash from Electric Power Production on Changes in Water Quality", with R.K. Guthrie. Water Resources Bulletin 17(6), 1981.
- "Fate of Chemical Elements from Coal Ash Correlated with pH of Effluent Water",
 Presented at the 5th International Biodegradation Symposium, Aberdeen, Scotland, 1981.
- 3. "Pretreatment Through Chemical Oxidation", Presented at the 40th Annual Purdue Industrial Waste Conference, May 14-16, 1985.
- 4. "Preimpoundment investigation of Water of the Bend Reservoir Area E.M. Davis, Principal Investigator. TDWR Contract no. 14-90030, August 1979.

Significant Technical Projects

- 1. "Catalytic Oxidation of Waste Sludges" (1981). (Internal Interox study)
- 2. "Study of H₂0₂ as an Oxygen Source for Bioreactors" (1982). (Internal Interox study)
- 3. "Stabilization of H₂0₂ Solutions" (1983) (Internal Interox study)
- 4. "Chemical Oxidation of Industrial Wastes" (1984 -1986).
- 5. "Peroxygen Compounds in Organic Synthesis" (1986-1990).
- 6. "Process Hazards of Active Oxygen Substances in Chemical Processing" (1988-1990).

Related Project Experience

NOTE: AP = Active Participant (project input, field sampling, data analysis, report writing)

PM = Project Manager (scheduling, client interface, data management, cost management)

OS = Oversight (design/protocol, budgeting, quality assurance)

Projects in *italics* designate ongoing commercial applications.

Municipal Sulfide (Odor) Control

Role	Client/Customer	Application (Technology)
AP	Ft. Worth, TX - Village Creek (1980)	Gravity interceptor collection system (H ₂ O ₂)
AP	Washington, DC - Blue Plaines (1980)	Solids processing (Fe + H_2O_2)
AP	Allentown, PA (1981)	Solids processing (Fe + H_2O_2)
ΑP	West Palm Beach, FL (1982)	Gravity interceptor collection system (H2O2)
AP	San Diego, CA (1984)	Odor scrubber (caustic + H ₂ O ₂)
PM	Wilmington, NC (1991-2)	Force main collection system $(H_2O_2 + KMnO_4 + pigging)$
PM	New Hanover County, NC (1992)	Force main collection system (H ₂ O ₂ + NaNO ₃)
PM	Wrightsville Beach, NC (1992)	Force main collection system (H2O2 + pigging)
AP	N.E. Monmouth, NJ (1991)	Mixed collection system (NaNO3 + H2O2 + KMnO4)
OS	Stockton, CA (1994-5)	Mixed collection system (NaClO ₂ + H ₂ O ₂)
OS	Jacksonville, FL (1994-6)	Force main collection system (NaClO ₂ + H ₂ O ₂ + NaNO ₃ +
		ClO ₂ shocking + Sulf Control ^m shocking)
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OS	Camden Co., NJ (1994-6)	Mixed collection system (NaNO3 + H_2O_2)
PM	Pittsburgh, PA (1994)	Solids processing (NaNO ₃ + H_2O_2 + FeCl ₃)
OS	Glendale, AZ (1994)	Gravity collection system (H2O2)
OS	Orange Co., CA (1994)	l) Headworks (H2O2)
	16 1 d	2) Odor scrubbers (caustic + H ₂ O ₂)
OS	Mukilteo, WA (1994)	Force main collection system $(H_2O_2 + NaNO_3)$
os	MWRA (Boston, MA - 1994-5)	Collection system (H ₂ O ₂)
OS .	Everett, WA (1994)	Collection system (H2O2)
OS	Newport, OR (1994)	Collection system (H ₂ O ₂)
PM OS	Chicopee, MA (1995)	Solids processing $(NaNO_3 + H_2O_2 + FeCl_3 + NaClO_2)$
OS	Hull, MA (1995)	Solids processing (NaNO ₃ + H_2O_2 + $FeCl_3$ + $NaClO_2$ + $KMnO_4$)
OS	S.Seminole Co, FL (1995)	Force main collection system (NaClO ₂ + H_2O_2 + NaNO ₃ +
00	5.5emmore Co, 1 2 (1775)	ClO ₂ shocking)
OS	Unified Sewerage, OR (1995)	Force main collection system (NaNO3 + H2O2 + FeCl2)
OS	Fox Metro, IL (1995)	Force main collection system (NaNO3 + NaOH shocking)
OS	Dayton, OH (1995)	Collection system survey (H2O2)
PM	Fresno, CA (1995)	Headworks (H2O2)
OS	Burlington, MA (1995)	Collection system (NaNO ₃ + H ₂ O ₂)
OS	S. Essex (Salem), MA (1996)	Headworks (NaClO ₂ + H_2O_2)
PM	San Jose, CA (1996)	Headworks (H2O2)
PM	Los Angeles, CA (1997)	Headworks and odor scrubbers (H2O2)
PM	Encina, CA (1997)	Headworks (H2O2)
PM	Monterey, CA (1999)	Headworks ($FeCl_3 + H_2O_2$)
PM	Foster City, CA (1999)	Collection system (H2O2)
PM	Oceanside, CA (2000)	Headworks (H2O2)
PM	Los Angeles, CA (2000)	Collection system (H2O2 + NaOH- H2O2 fogging)
PM	Orange County, CA (2001)	Collection system (FeCl ₂ + H_2O_2)
os	Redding, CA (2001)	Collection system (H2O2)
OS	San Francisco, CA (2003)	Collection system (FeCl ₂ + H_2O_2)
Industr	ial Sulfide (Odor) Control	
4.70	GI 11 O'I 37 P G (1999)	
AP	Shell Oil - Norco Refinery (1982)	Sour water stripper bottoms (catalytic H ₂ O ₂)
AP	Tenneco Chemical - Pasadena, TX (1983)	Pond turnover (Na ₂ CO ₃ + H ₂ O ₂)
PM <i>PM</i>	Chevron Oil - Texas City Refinery (1984)	Sour water filter backwash (Fe + H ₂ O ₂)
PM PM	Chevron Oil - Pascagoula Refinery (1984)	Pond turnover (Na ₂ CO ₃ + H ₂ O ₂)
F IVI	Mobil Oil - Torrance Refinery (1984 - 85)	1) Sour water stripper bottoms (catalytic air + H_2O_2)
		2) API separator effluent (Fe + H ₂ O ₂)
PM	Golden West Refinery (1984)	3) Slop oil tank clean-out (NaClO ₂)
PM	Texaco Oil - Wilmington Refinery (1985)	Misc. process waters ($Fe/H_2O_2 + ClO_2$) Spent caustic (catalytic air + H_2O_2)
AP	Texas City Refinery (1985)	MEA-DEA absorbent (H_2O_2)
AP	ChemTreat (1986)	From sponge regeneration natural gas (H_2O_2)
PM	Merichem (1986)	Spent caustic (H ₂ O ₂)
РМ	SCM-Glidco (1987)	Crude sulfated turpentines (catalytic H2O2)
PM	Chevron Minerals (1991)	Uranium extraction off-gasses (NaClO ₂ + counteractant)
PM	Champion Paper - Canton, NC (1991)	Solids dewatering (KMnO ₄ + FeCl ₃ + H ₂ O ₂)
AP	USPCI (1992)	Petroleum sludges (KMnO ₄)
AP	Nevada Power (1992)	Waste gypsum settling pond (KMnO ₄)
PM	K&E Steel - Toledo Plant (1992)	Slag quench water (catalytic air)
PM	Syntex Paper - Pomona (1992)	Wastewater clarifier (KMnO4)
OS	Chevron Oil - El Segundo (1992)	Reactor clean-out (KMnO4)
		. "

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PM	A.E. Staley (1993)	Wastewater collection system (H ₂ O ₂)
PM	North Star Potato (1994)	Digester decant (Fe + H_2O_2)
OS	California Spray Dry (1994)	Rendering plant off-gasses (ClO2 scrubber)
OS	Corn Products - Stockton (1994)	Wastewater collection system (H ₂ O ₂)
OS	Jay Dee - Healy (1995)	Construction groundwater (NaClO ₂ + catalytic air + H_2O_2)
OS	Waste Recovery Systems (1995)	Waste food oils (percarbonate + NaClO2)
OS	IVEX Paper (1995)	Wastewater clarifier (H ₂ O ₂)

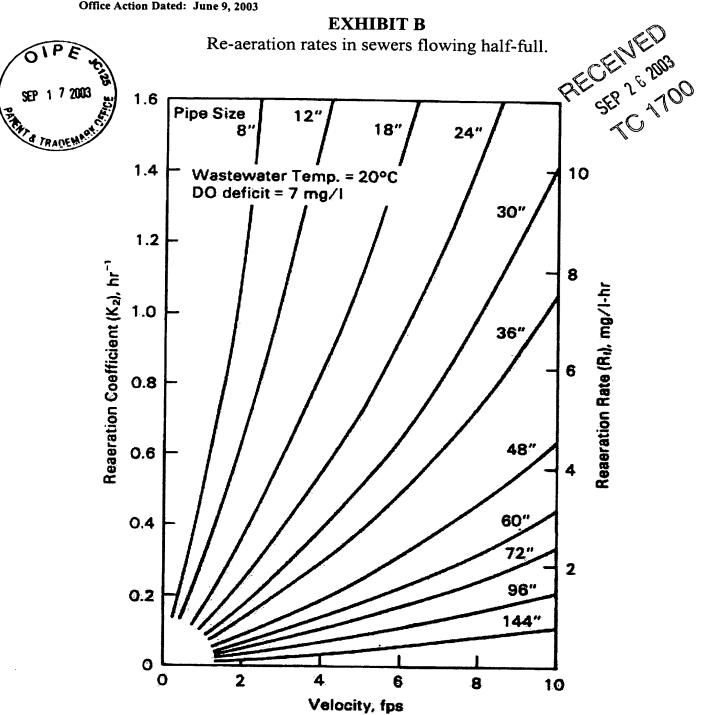


Figure taken from "Design Manual: Odor and Corrosion Control in Sanitary Sewerage Systems and Treatment Plants", EPA/625/1-85/018, October 1985, Figure 2-7, page 12.